

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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| In re Application of : | § | Before the Examiner |
| Zhao et al. | § | James E. McDonough |
| Serial No. 10/693,584 | § | Group Art Unit No. 1793 |
| Filed: October 24, 2003 | § | Attorney Docket Number: 2002B130/2 |
| For: Late Transition Metal Catalysts for Olefin Oligomerizations | § | Confirmation Number 1077 |
| | § | |
| Customer No.: 23455 | § | Date: July 18, 2008 |

Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Declaration Under 37 C. F. R. § 1.132

Dear Sir:

This affidavit is submitted to offer a showing that Claims 2-6 and 9-16 are both novel and non-obvious over Buchwald (US 6,307,087) in view of Qian (*Synthesis and Polymerization Behavior of Various Substituted Half-Sandwich Titanium Complexes Cp'TiCl₂(OR*) as Catalysts for Syndiotactic Polystyrene*, J. Mol. Cat. 208, 2004, 45-54.).

This declaration is submitted with a response to the Advisory Action dated July 7, 2008, along with an Information Disclosure Statement, as the submission required under 37 CFR § 1.114 for a Request for Continued Examination.

I, Jo Ann Marie Canich, Ph.D. declare that:

1. I am a citizen of the United States residing in Houston, Texas.
2. I am a co-inventor of the above-referenced patent application referred to herein as the '584 Patent Application.

3. I have been continuously employed by ExxonMobil Chemical Company, (and its predecessor Exxon Chemical Company) since 1987 as a research scientist working in the area of olefin catalysis. I have over twenty years of direct experience in the olefin catalysis art and, among other things, have recently published a book (co-editor) in the area of olefin catalysis (*Stereoselective Polymerization with Single-Site Catalysts*, Eds. Baugh & Canich, CRC Press, Boca Raton, FL, 2008). I have won multiple awards both inside my company and from industry for my work in the olefin catalysis field. Additional information regarding my technical and professional background is attached as a *Curriculum Vitae*.

4. I have read the Office Action dated February 22, 2008 and Advisory Action dated July 7, 2008, and have read each of the references cited therein, e.g. Buchwald and Qian.

5. In my opinion one of ordinary skill in the olefin catalysis art reading Buchwald would find that Buchwald is directed to catalysts useful to make small molecules via metal mediated coupling reactions, but not polymerization or oligomerization catalyst compounds. Furthermore, it is my opinion that the reference to "polymers" in Column 1, lines 16-20 is merely a general opening statement regarding the uses of transition metal catalyst compounds. In this sentence located in the "background of the invention" section, Buchwald in no manner implies that the catalysts of his invention are polymerization catalysts. In fact, in searching for the word "polymer", or a derivative of the word polymer, only 6 occurrences are found in the entire 55 page document. The second occurrence is on Column 16, line 56 where Buchwald again has a general statement regarding the use of synthetic schemes for aryl amines, biaryls, alpha-aryl ketones, and the like being useful to the agricultural, pharmaceutical and polymer industries. Again, this is simply a general statement, and in my opinion suggests that the coupling chemistry described in this patent can be used to add functionality to a polymer at most, i.e. performing coupling chemistry *with* polymers, not using coupling chemistry to *make* polymers. Furthermore, there is no indication, illustration or exemplification in the entire document that the coupling chemistry can be used to make polymers. Further in my opinion, even if one were to stretch one's imagination to the concept of using the catalysts for making polymer, one would be limited to making polymers containing aryl groups. All four reactions schemes discussed in the patent (see column 10, line 5, column 12, line 5, column 13,

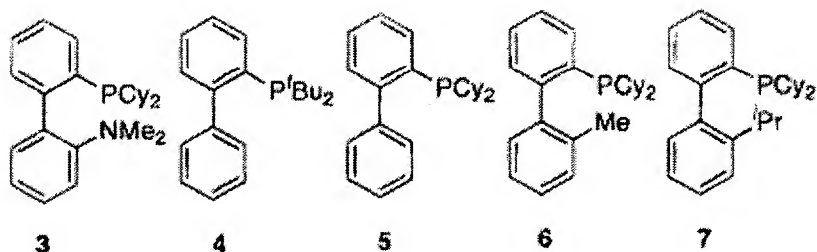
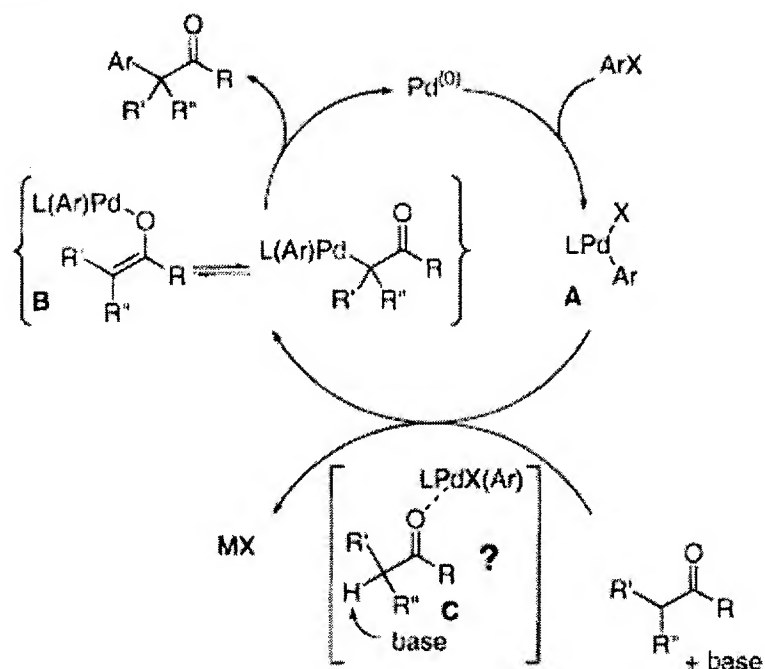
line 30, and column 14, line 60) and also claimed (see claims 19, 38, 50, and 62) involve the coupling of an aryl group to either an amine (Scheme 1), another aryl group (Scheme 2), an alkyl, heteroalkyl, or aralkyl (Scheme 3), or a ketone (Scheme 4). There is no mention or teaching of coupling an alpha-olefin with a second alpha-olefin, and there is certainly no mention of stringing more than two of any molecules together in any of these reaction schemes.

Besides the two occurrences of “polymer” discussed above, the remaining four are clearly related to polymer supports regarding supported Pd catalysts or polymer supported reactants which are typically used in combinatorial chemistry.

6. It is further my opinion that Buchwald does not disclose the pre-catalyst compounds or polymerization catalysts used in my patent application, and only discloses the use of the ligand in combination with a metal and a base such as an alkoxide, amide, phosphate, or carbonate. While the Examiner suggests that the reaction of the ligand with a metal will produce the pre-catalysts of my invention, this is not indicated in or supported by Buchwald.

In fact, if one refers to *Journal of the American Chemical Society* 2000, 122, 1360 (Fox et al.), Fox et al. shows the proposed reaction scheme (Scheme 1, page 1366 - also shown below) for a small molecule coupling reaction where the ligand, L, is shown in the chart on page 1362. The ligand labeled **3** can perform as an ancillary ligand where Y is biphenyl (referring to the formula in Claim 1 of the ‘584 application). On page 1366, Fox et al. states that in comparison to previous studies, that they believe the Pd:phosphine ratio when using ligands **4-7** for the reaction intermediates (**A** and **B**) is 1:1 (i.e. only one electron donating atom, in this case phosphorous, is needed). Note that ligands **4-7** are mono-dentate phosphorus ligands. Additionally, similar evidence suggests that the binding of the dimethylamino group of ligand **3** to Pd is not essential for the catalytic process and, in fact, may not occur at all. This is indicated by the similarities in the catalysis involved when using **3**, **6** and **7** in the Pd-catalyzed ketone arylations reported in the paper, in addition to Suzuki and amination reactions reported in references within. Note that ligands **6** and **7** do not contain the dimethylamino group as compared to ligand **3**. Hence, it would be speculative at best to assume that the reaction between ligand **3** and a metal would form the polymerization pre-catalyst of my invention, when the chemistry in small molecule coupling reactions suggests otherwise.

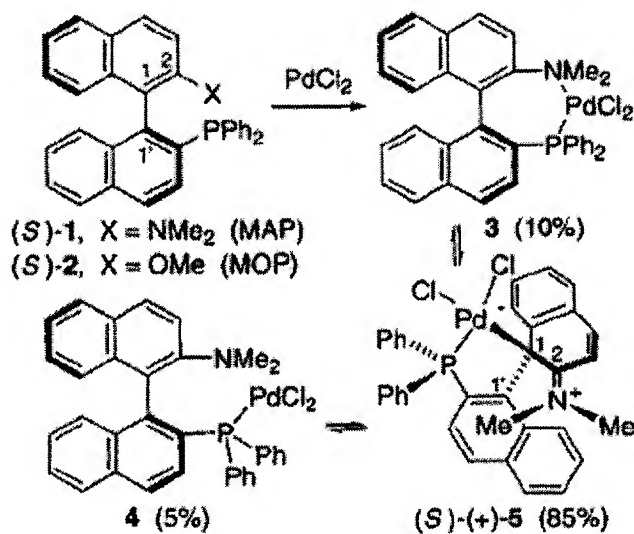
Scheme 1



Fox et al. also states that it is possible that the interaction between the metal and the ligand in the Pd(II) intermediates complexes (**A** and **B**) is similar to those reported for the MOP and MAP ligands reported in *Journal of the American Chemical Society* 1999, 121, 7714 (Kocovsky et al.). Kocovsky et al. uses a binaphthyl ligand (not an ancillary ligand in my application). Scheme 1 of Kocovsky et al. is shown below (p 7714). Kocovsky et al. indicates an equilibrium between species **3** (10%), **4** (5%) and **5** (85%) in solution, however, they were only able to isolate compound **5**, suggesting the preferred state of the molecule is complex **5**. This is based not only on the ability to isolate complex **5** in crystalline form which was structurally characterized by X-ray crystallography, but also on the solution equilibrium mixture

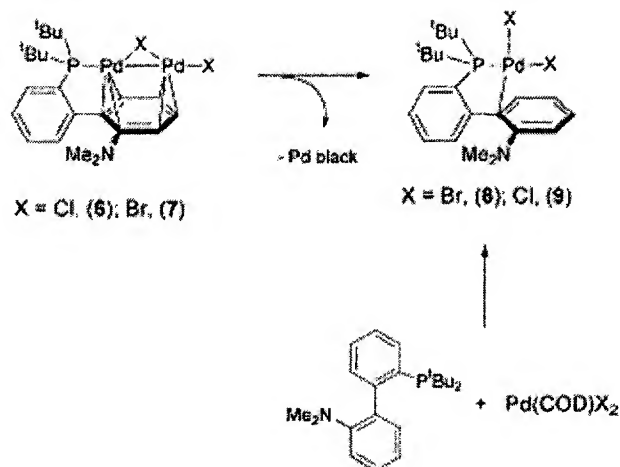
being 85% complex **5**. Note that complex **5**, and also complex **4**, does not have a metal-nitrogen bond as required of my ancillary ligand in the claims. Again, it would be speculative to assume the reaction between the ligand of Buchwald would react to make the complexes of our application verses assuming other chemistry as suggested by Kocovsky.

Scheme 1



Also noteworthy, is *Journal of the American Chemical Society* 2006, 128, 6376 (Christmann et al.) where reaction Scheme 3 illustrates two routes in making a palladium dihalide complex bearing a 2'-(di-tert-butylphosphino)-*N,N*-dimethylbiphenyl-2-amine ancillary ligand. Note that the reaction of the ligand with $\text{Pd}(\text{COD})\text{X}_2$ where X is chlorine or bromine and COD is cyclooctadiene produces the Pd complexes labeled **8** and **9** wherein the Pd metal center is not bonded to the amine functionality of the ancillary ligand. Christmann et al. characterized complex **8** by X-ray crystallography, and both complexes were characterized by NMR.

Scheme 3



One should also note that the catalysis scheme for making small molecules via metal mediated catalysis as shown in Fox et al.'s Scheme 1 involves (1) starting with a Pd(0) complex and oxidizing it to Pd(II) by ArX to form intermediate **A**; (2) forming an interaction between intermediate **A** and an electron donor (a ketone in this case) in the presence of a base to form intermediate **C**; (3) abstraction of an acidic proton by the base to form intermediate **B**; and finally (4) reduction of intermediate **C** (Pd(II) down to Pd(0)) and formation of an arylated ketone. This is a catalytic cycle that involves multiple Pd species, and requires the ability to oxidize the metal to a higher oxidation state in order for the chemistry to occur, and then reduce the Pd back to Pd(0) to regenerate the catalyst and to "release" the new compound. The coupling mechanism for Buchwald is analogous to that represented by Fox et al. This is why Buchwald states in Column 32, line 7-8 that "the metal center is desirably in the zero-valent state or is capable of being reduced to metal (0)". The nickel and palladium species in my application are Ni(II) and Pd(II) and the catalytic cycle for olefin polymerization/oligomerization does not involve changing the oxidation state of the catalyst precursor, or of the catalyst species during the polymerization / oligomerization reaction.

7. Also interesting and noteworthy is that only nickel complexes bearing the 2'-(*dihydrocarbyl*-phosphino)-*N,N*-*dihydrocarbyl*-biphenyl-2-amine ancillary ligand recognized to date (July 11, 2008) by the Chemical Abstract Service (CAS) are [2'-(diphenylphosphino)-*N,N*-dimethylbiphenyl-2-amine]nickel(II) dibromide and [2'-(dicyclohexylphosphino)-*N,N*-dimethylbiphenyl-2-amine]nickel(II) dibromide

bearing registry numbers 685517-32-2 and 685517-31-1, respectively. The only references to these compounds are WO 2004/037869 and WO 2004/037837 which are related to my US '584 application. Also, while CAS now includes prophetic compounds in the registry as of Dec 12, 2007, these registry numbers and references are prior to the implementation of including prophetic compounds. Thus another in the art (i.e. the CAS) only recognized my art in the above referenced patent applications, and specifically not Buchwald, to have made the metal complexes as claimed in my '584 application.

8. It is further my opinion that Qian is directed to polymerizing styrene, not to oligomerizing or polymerizing alpha-olefins as in my application. As such, a trend seen in Qian can not necessarily be equated with a different type of polymerization process. Qian is focused on changing the Cp' or R* of Cp'TiCl₂(OR*). Qian states, "In order to study how the changes of pi-donor ligand and sigma-donor ligand affect the catalytic activity and the properties of s-PS, this work aims to design and prepare a series of Cp'TiCl₂(OR*) complexes with different substituents in Cp and OR*, and then to apply them to the production of s-PS." Since Cp'TiCl₃/MAO is known to be a styrene polymerization catalyst, perhaps the Examiner is comparing the OR* of Qian to Cl, but OR* is not a hydrocarbyl, nor is it an abstractable ligand/leaving group in this catalyst system. The conclusion in Qian states, "through the polymerization testing we can conclude that the environment of cyclopentadiene plays a major role in the series of Cp'TiCl₂(OR*) /MAO catalytic systems, and that the influence of OR* is relatively weak." Qian's conclusion does not mean that R* has little effect, it means that in comparison to changing the substituents on the Cp ring, it has little effect for the polymerization of styrene.

Furthermore, it is commonly known in polymerization catalysis that changing the leaving group can affect catalyst activity, polymer molecular weight, and when applicable, polymer tacticity. For example, Kaminsky et al. reported that the styrene polymerization activity of fluorinated complexes Cp'TiF₃/MAO (Cp' = Cp, Cp*, MeCp, EtMe₄Cp, etc.) was about 30-40 times higher than with analogous Cp'TiCl₃/MAO systems. The polymer molecular weight, melting point and syndiotacticity were also significantly higher. This higher activity and syndioselectivity of the fluoride catalysts as compared to the chloride analogs is attributed to a greater number of more stable Ti(III) active sites where the fluoride

would be expected to better stabilize the Ti(III) oxidation state. See *Macromolecules* 1997, 30, 7647-7650. Similarly, in ethylene polymerization, McCullough et al. in US 6,632,901 report higher activity with $\text{Cp}'_2\text{ZrF}_2$ is used as compared to $\text{Cp}'_2\text{ZrCl}_2$ ($\text{Cp}'=1,3\text{-Me,BuCp, Me}_4\text{Cp, n-PrCp, etc.}$) Similarly, Pedeutour et al. (*Journal of Molecular Catalysis A: Chemical* 2001, 176, 87-94) studied the effect of the leaving group for $\text{rac-Et(Ind)}_2\text{ZrX}_2$ where X, the leaving group, was Cl, Me, NMe_2 or CH_2Ph . In this study, it was found that when X was hydrocarbyl (Me or CH_2Ph) that catalyst activity was not dependent on the MAO to pre-catalyst molar ratio (Al:Zr) over the range of about 150-2000. This was not the case for the Cl leaving group where the activity differed significantly from low Al:Zr of 150 (activity = 0) to a higher Al:Zr of 2000 (activity = 1290 kg/mol-hr). This study clearly shows that for a polymerization catalyst leaving group, hydrocarbyl is not equivalent to halide. Probably more relevant is US 7,247,687 (Cherkasov et al.) which deals with late transition metal polymerization catalysts. Cherkasov et al. demonstrate that the [1,4-bis-substituted)-1,4-diaza-1,3-butadiene]nickel(II) catecholate complexes are very different catalysts as compared to the dibromide analogs. Note that the two differ in the X leaving groups - two bromides vs. a chelating catecholate ligand. The dibromide complexes are known paramagnetic and typically hydrocarbon insoluble complexes while, the catecholate analogs are diamagnetic and hydrocarbon soluble (see column 2 for a discussion on paramagnetic complexes in the Background section and diamagnetic complexes in the Summary section). Comparison of the polymerization of a catecholate based catalyst vs. corresponding dibromide based catalyst (compare entries 8-11 with C6-C7 in Table 1, column 308) shows the catecholate to give higher activity as compared to the dibromide. While this study did not compare hydrocarbyl to halide, it does show that the nature of the leaving group affects catalyst activity in late transition metal catalyzed olefin polymerizations.

Since many examples exist to show that the leaving groups in olefin polymerization catalysis can have a major affect on catalyst activity, one skilled in the art would not assume that a hydrocarbyl leaving group is the same as a halogen leaving group in a nickel based catalyst system.

9. It is my further opinion that halogen ligands and hydrocarbyl ligands are not equivalent in all catalyst systems at all times. First halogen ligands are considered electron withdrawing, whereas hydrocarbyl ligands are typically

considered electron donating. Second, in many systems, a borate activator in combination with a halogenated catalyst precursor (e.g. Cp_2ZrCl_2) produces an inactive system while an alkylated precursor (e.g. Cp_2ZrMe_2) will likely, but not always, produce an active system. Thus, halogens and hydrocarbyls are not equivalent, particularly in borate activator containing systems.

10. In light of this lack of equivalence and the other statements above, it is my opinion that one of ordinary skill in the art would not combine Buchwald and Qian because firstly Qian is not directed at comparing hydrocarbyl verses halogen leaving groups, but is a study of ligand substitution on mono-cyclopentadienyl titanium catalysts for styrene polymerizations; and secondly, Buchwald is not directed to olefin polymerization catalysis, but rather to small molecule synthesis using late transition metal mediated coupling chemistry.

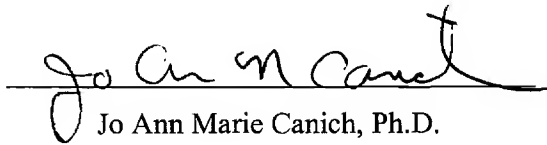
11. At the time the '584 application was filed, very few complexes used in catalyst systems were known that combined good catalyst activity with the production of linear olefins, with a particular preference for lower alpha-olefins. Additionally, the hydrocarbon solubility of my complexes (i.e. catalyst precursors) allowed for readily supporting the complexes to produce supported catalysts which can have advantages in certain processes, for example, a fixed bed process. And it is possible (but yet unproven) that the reason behind the unusual catalysis of my catalyst system, is that most four-coordinate Ni(II) complexes prefer the planar geometry since the planar ligand set allows for the d-electrons to avoid occupying antibonding orbitals. My Ni(II) four-coordinate complexes were determined to be tetrahedral in geometry meaning that d-electrons occupying the antibonding orbitals is unavoidable, hence the fact that these complexes are also paramagnetic.

12. In light of the above, it is my opinion as one of ordinary skill in the art that the foregoing clearly indicates that the catalyst described and claimed in my '584 Patent Application represents a technological break-through as perceived by those skilled in the field of transition metal catalysis.

13. *I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both,*

under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the patent.

Date: July 18, 2008


Jo Ann Marie Canich, Ph.D.

CURRICULUM VITAE

JO ANN MARIE CANICH

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Industrial Positions:

- ExxonMobil Chemical Company – Core Technology
 - Product Technology, Organometallic Catalysis Group (11/07-present)
 - Team Leader - Block Copolymers Team 1/08-present
 - Catalysis Process R&D, Organometallic Catalysis Group (1/07-11/07)
- Univation Technology: Catalyst R&D (1/04-1/07)
- Exxon or ExxonMobil Chemical Company – Core Technology
 - Catalysis Process R&D, Organometallic Catalysis Group (6/02-12/03)
 - Intellectual Property Coordination (12/02-12/03)
 - Polymer Science Division, Catalysis & Process Science Group (5/99-5/02)
 - Polymer Science Division, Solid State and Molecular Modeling Group (1/96-4/99)
 - Team Leader - Exploratory Catalyst Team 1/96-12/97
 - Team Leader - Solution Metallocene Elastomers Team (1/98-12/98)
 - Exxpol Catalysis Group (2/93-1/96)
 - Polymer Science Division, Catalysis Group (9/87-2/93)

Awards:

- ExxonMobil Chemical Company - CPR&D Research Award 2003
- American Society of Patent Holders Distinguished Corporate Inventors 1998
- Exxon Chemical Polymers Technology Award 1997
- Exxon Chemical, Baytown Polymers Center, Extra Mile Award 1995
- Exxon Chemical, Polymer Science, Where the Tire Meets the Road Award 1990
- Iota Sigma Pi National Anna Louise Hoffman Award 1987

Education:

8/84 - 5/87 Texas A&M University
College Station, Texas
Ph.D. Inorganic Chemistry (8/87)
Advisor: F. A. Cotton
Thesis: Oxidative Addition Chemistry of Group V and VI Metal Dimers

9/77 - 7/84 Portland State University
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M. S. Chemistry (6/84)
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Advisor; G. L. Gard
Thesis: Derivatives of Trifluorosulfur Pentafluoride and F-(tert-butyl)
Hypochlorite

Publications:

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